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CARBIDE AND CARBON CHEMICALS CORPORATION

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POST OFFICE BOX P OAK RIDGE, TENN.

February 20, 1947

J.C. NO 3608

U. S. Atomic Energy Commission Post Office Box E Oak Hidge, Tennessee

Attention: Lt. Col. R. W. Cook

Dear Sir:

Attached is a report on (the recovery/and disposal of radioactive material in plant waste at K-25 which is submitted in answer to the request of your office on 5 December 1946.

Very truly yours,

C. N. Rucker, Jr.

Assistant Plant Superintend

C.N.Rucker, Jr. RWL/jw

Lt. Col. R. W. Cook Copies:

C. E. Center

R. W. Levin

C. M. Rucker, Jr., file

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CLINTON ENGINEER WORKS CARBIDE AND CARBON CHEMICALS CORPORATION Uranium Control and Inspection Department

3608

February 20, 1947

Recovery and Disposal of Radioactive Material in Plant Waste at K-25

R. W. Levin

Introduction

A discussion of the recovery and disposal of radioactive material in plant waste at K-25 is largely concerned with uranium contaminated material. There are other radioactive materials than uranium at K-25 such as radium-beryllium neutron sources but these are so few in number and only rarely produce radioactive wastes that they constitute no major problem at this time.

The operation of the gaseous diffusion plant produces small amounts of uranium waste in the form of solutions, oils, carbon and alumina, and scrap metal. In addition there is a constant deposition of uranium compounds on the metal surfaces in the cascade.

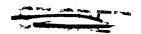
A general flow diagram of materials contaminated with uranium is given in Figure I. This shows the major recovery problems to be:

- Recovery of uranium from metal surfaces removed from process gas service.
- 2. Recovery of uranium from oils.
- 3. Recovery of uranium from carbon.
- 4. Recovery of uranium from activated alumina.
- 5. Recovery of uranium from water media.

The extent of recovery or removal of uranium from these materials and the disposition of the wastes should be based on several factors. These are cost of recovery, health hazards due to insufficient removal of uranium, accountability, and diversion control.







Status of Recovery Problems

Recovery of uranium from metal surfaces removed from process gas service

There is a fairly constant deposition of uranium compounds on the metal surfaces of the cascade. The estimated rate of this consumption has been reported by Mr. J. P. Kelly, "Consumption Estimates", A-3666, January 10, 1947. Thenever equipment is removed from process gas service it is necessary to remove the small amount of uranium on the metal surfaces before the equipment can be safely serviced or repaired. There are two general methods of removing the deposits:

- 1. Washing the equipment with an appropriate solution.
- 2. Fluorinating the equipment with F2 to form volatile TF6.

The first method is used by Cascade Services Decontamination Unit in K-1303 and is described in detail in the Process Division Standard Operating Procedures, series 900. Completeness of decontamination is determined by moistening a test paper with 1% potassium ferrocyanide solution and rubbing the paper over several surfaces of the item decontaminated. This test shows the absence of any soluble uranium compounds, but does not detect insoluble compounds such as TF4.

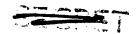
The second method is used almost exclusively for recovery of uranium from converters for consumption studies and is carried out in K-1401. Completeness of fluorination is determined by gas analysis.

Solutions obtained from the Decontamination Unit are sent to a contaminated storage lot pending determination of the desirability of recovery. Converters removed from process gas service are not routinely fluorinated, but are placed in storage without recovery. Equipment which has been decontaminated is then repaired for use, placed in storage, or declared surplus or scrap. Surplus or scrap material is not examined for radioactivity at the present time since the amount of radioactivity from uranium compounds left on the surfaces is well below tolerance for personnel safety. However, an investigation is being made of procedures for use in determining the absence of low level radioactivity.

Recovery of uranium from oils

There are two types of oils which become contaminated with uranium. The first is special fluorinated oils and the second is regular hydrocarbon oils. During the past year the amount of these oils in process gas service has been greatly reduced, and only very small quantities of contaminated oils are produced. About three fourths of the backlog of contaminated oils has been reworked for uranium recovery. The decontaminated oil is then sent to storage for reuse; however, a process for reclaiming the uranium from the oil filter cake has not been developed, and the filter cake is being stored.





Recovery of uranium from carbon and alumina

During the early operation of the plant, large amounts of contaminated carbon and alumina were produced, but during the past year the number of traps in active service has been appreciably reduced so that normally less than 200 pounds of uranium per month is trapped in carbon and alumina. The Engineering Development Division is developing methods of recovery of uranium from carbon and alumina, and an appreciable fraction of the backlog of contaminated carbon has been reworked in the process of developing satisfactory methods.

Recovery of uranium from water media

Solutions obtained from decontamination of equipment and spills, and from laboratory or experimental wastes are sent to contaminated storage prior to recovery of the uranium as an oxide and subsequent conversion to TFg. The desirability of recovering uranium from waste solutions or carbon and alumina for use as a feed to the cascade should be considered on the basis of economy of operation as well as accountability and diversion control. The cost of recovery can be compared with the value of the increased cascade production due to the additional feed of recovered TFg. A report recommending limits for discarding contaminated waste solutions and carbon on this basis has previously been issued to Lt. Col. R. A. Cook on January 16, 1947.

Inventory of Radioactive Wastes Stored at the Plant, and Amount of Waste Reworked

Tables I - 3 show the amounts of waste reworked, amounts of uranium recovered, and the inventory of uranium bearing waste material on 31 December 1946. The largest uranium inventory in waste is in spent carbon and nearly all of this is feed concentration or less.

Figures 2 - 10 are flow sheets and descriptions of the processes used in reworking waste materials.

Acknowledgement

Figure I and Tables I - 3 were prepared by Mr. F. Mills.
Acknowledgement is made for permission to use figures 2-10 prepared by
K-25 Division U.S.A.E.C.



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FIGURE II

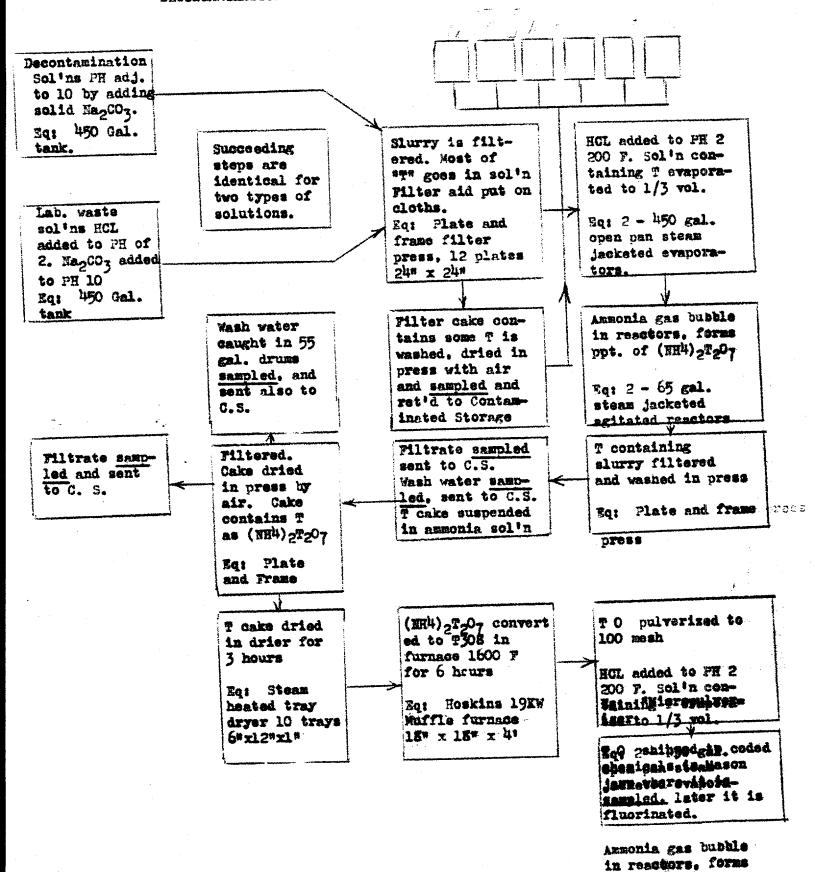
THE RECOVERY OF "I" FROM COMTANINATED "ATERIAL

	CONTAMINATED STORAGE	ann i M
Con	taminated Solutions	A STATE OF THE STA
(a)	and the second s	Pecontamination Carbonate
(a)	decontaminated in place	water, or Hydrogen peroxide
	Gecontaminated in brace	3 (n. 4 d
		Carbonate solutions, but may be
(も)) Experimental users' maste	Carponavo Bordon no por
		lany tyre Vlab. aste sclutions
(c) Laboratory waste	Miab. aste collections
, ,	•	
(ব) Jeak Solutions from recovery process	es Shinned to contaminated
(()	(stored)	
		Contaminated equipment heated
2 ქი	ntaminated Equipment	In dilute NeoCO, solin. Solin
(a) Residue in cylinders	and sludge contain "T" Four
(b	 Fumps and Seals Valves and pipes Gloves and clothing Line recorder cold traps 	Classified tanks used and one
(c) Valves and pipes	Volassined camp used and
6)) Gloves and clothing	wash tank. Sampling - C.D.,
	1 Time recorder cold traps	and E tanks sampled Mon. Tues.
E V 78	The state of the s	Wed. Thur. All tanks sampled
	Contractors	A second of the
[[I	') Converters	3
:		1
3 F1	uorocarbons () Mixtures of TF6 and C-816 removed for cascade at or near 3C1-4	2000
; (a	() Mixtures of TF6 and C-old removed I	1 Opp
•	cascade at or near 301-4 Mixtures of TF6 and Freon and other fluorocarbons removed from cascade	hoo - withdrawn once and sent
(h)) Mixtures of TF6 and Freon and other	200 g. WICHGIAWH ONCO WHA DOLLAR
•	fluorocarbons removed from cascade	at to laboratory for analysis
, ,	306-7.6	
a co	nntnmingted Mil#	,,,
	MFL from Beach-Fuss pumps b) 2144 from valley iron numps With the company of the	2
(8	Old from rollow inch number	
\$ C	0) 2144 Irom valley from daily	\$ 1 1
	e i HAGLOGALDON OTIS (200100)	
5 Co	ontaminated Filter Cakes	y Develorment of Process
(8	a) Cake following MFL or 2144 recover;	nor complete
:	(stored)	Ator Compress
· (1	b) Cake from recovery of decontaminat	cn
;	(sol'ns (stored)	
6 C	arbon	\ \ \ \ \ \ \ \ \ \
-	a) Seal exhaust traps	
	b) Carbon trans in cold trap rooms	
		Process being developed .
	d) Portable carbon traps	į'
- <u>'</u>	e) Mobile carbon traps	ion e
•	(The carbon is emptied from the tr	aps
į	into 30 or 55 gallon drums)	
7 A	ativeted Alumina	
	(a) Alumina trans from 312 buildings	Land 8
	(b) Alumina traps from purge and produ	ect Process being developed 8
•	room	······································
	aminated Materials Storage samples mater	rial
Conte	eceived except in instances where the ar	nalv-
as re	eceived except in instances whole the election as for	l ows :
sis :	is known. Material is classified as for	
	-	
	A Normal	į
	B Normal - 2230001	ì
į	c 2230001 - 3480003	
	D 3480003- 2810001	
	E 2810001- 31220002	
1	L 31220002-11070002	
	The second secon	

CONTRACTOR OF THE PARTY OF THE

FIGURE 3

DECONTAMINATION SOLUTIONS AND LABORATORY MASTE SOLUTIONS



SOFET

FIGURE IV FLUORINATION OF T3 08 TO TF6

Some TF6 gets by Any remaining T cylinders and is is scrubbed out by TF6 frozen out in TEC cylinders, two 1308 fluorinated to TF6 by KOH sol'n in the caught in cold cylinders in treating with Ascrubbing tower trap. Is later series surrounded \mathbf{F}_{2} flashed back to by dry ice sol'n Eq: Scrubbing cylinders or tower 6" Diam. Eq: Four cylindwashed out with -ical reactors 12' long with MOH Na₂CO₂ sol^tn long 6" Diam. 3 trays 1/2" deep solin circulated. TF6 shipped to coded chemicals pure storage where material is sampled. KOH sludge KOH sol'n is caught in trap is drawn off, sampled cleaned about once and sent to C.S. every 6 months, sampled and sent to C. S. Eq: Sludge trap is tank 4'x3'x1'

FIGURE 5

RECOVERY OF "T" FROM CONVERTERS

Converter is reconditioned by recirculating F2 and G-74 thru converter in a closed system

Eq: Converter Conditioning stand Any TF6 formed plus waste gases pass thru a cold trap after conditioning where TF6 is trapped. Other gases pass thru. Original tests showed less than 0.0005 mol. 5
T in waste gases.
Eq: Cold trap 15^t leng 3th Diam.
- 150 F

TTO flashed bask from cold trap to "D" type cylinder surrounded by dry ice col'n.

Sent to Coded Chemicals where material is sampled.

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FIGURE 6

RECOVERY OF "T" FROM MIXTURES OF TF6 AND C-816 REMOVED FROM THE CASCADE AT OR NEAR 301-4

(THIS HAS BEEN ABANDONED IN FAVOR OF A STILL COLUMN NOW IN PROCESS OF DEVELOPMENT)

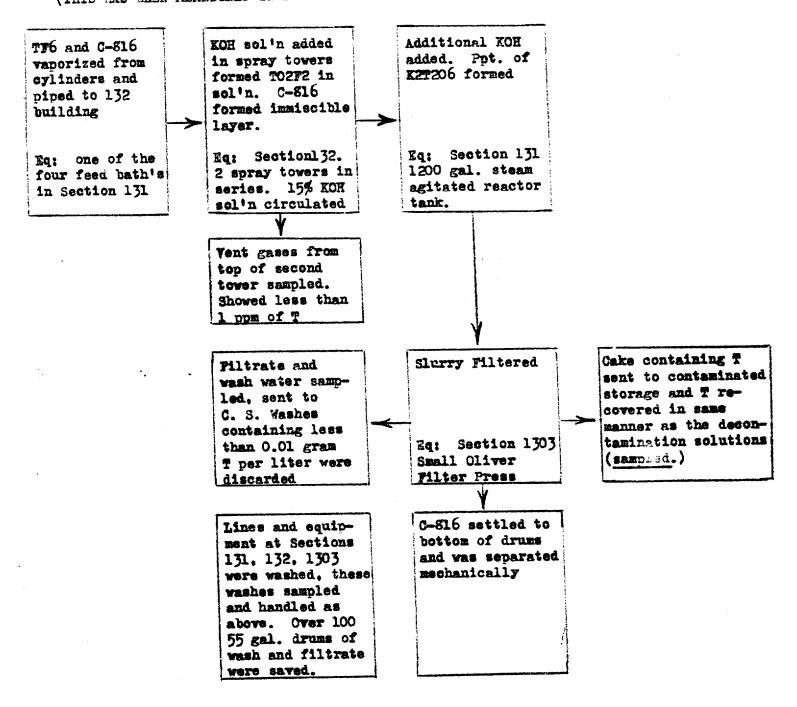


FIGURE 7

RECOVERY OF "T" CONTAINING CAKE FOLLOWING THE RECOVERY OF MFL

HOTE: RECOVERY OF "T" FROM CAKE HAS NOT BEEN DEVELOPED YET

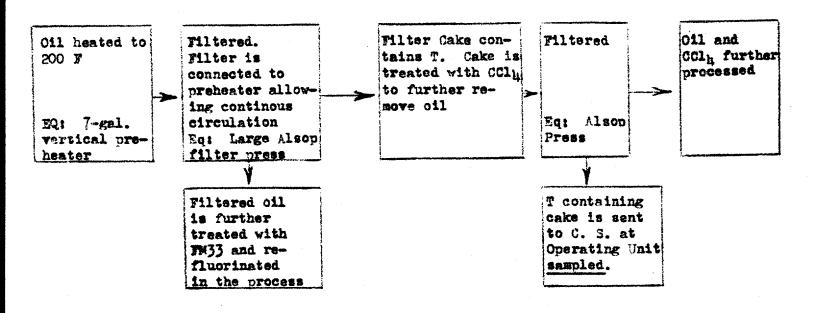


FIGURE 8

RECOVERY OF "T" CONTAINING CAKE FOLLOWING THE RECOVERY OF 2144

NOTE: RECOVERY OF "T" FROM CAKE HAS NOT BEEN DEVELOPED YET

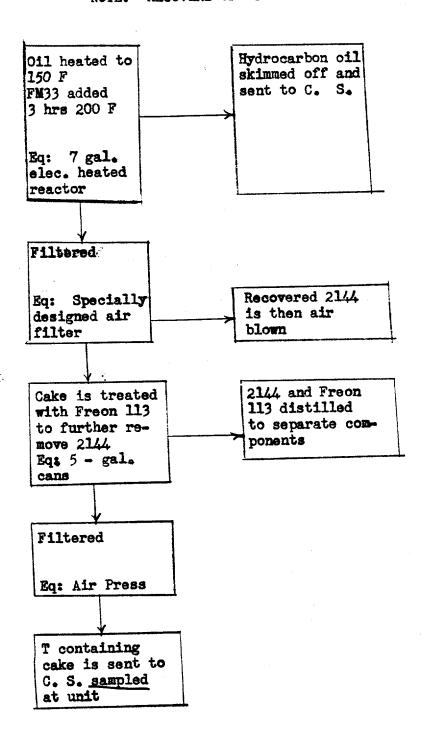


Figure 9

RECOVERY OF "T" FROM CARBON FROM CARBON TRAPS
THIS PROCESS IS BEING DEVELOPED AT THE PRESENT

The steps in the process are:

- 1. The separation of the alumina from the carbon. This alumina is being reused in making fresh carbon-alumina mixes.
- 2. The conversion of the T to T308 in a furnace.

Sampling

The probable sampling points after the process is developed will be the carbon-alumina-T mixture, and the converted T308.

Figure 10

RECOVERY OF "T" FROM ACTIVATED ALUMINA FROM ALUMINA TRAPS
THIS PROCESS IS BEING DEVELOPED AT THE PRESENT

The steps in the process will most likely be:

- 1. Treatment of alumina and T with Sulphuric Acid, followed by diluting and treating with Mitric Acid.
- Filtration. Filtrate contains the T, and will be handled the same as decontamination solutions.

Sampling

Probable sample points after the process is developed, will be the alumina and T mixture, filtrates, washes, and precipitates.

TABLE 1

ESTIMATION OF RECOVERED TF6 FROM REFORKED "FATER MEDIA" AND 816-616 MIXTURE AND THE BY-PRODUCTS FROM MAY 1946 TO JAM, 1947

Unnocounted	Table 2	Charged to Recovery From	Total nin in Nasto	พorked	Total Wiff Completely 10-	Monorked except TI6	Concensor	Filtrate from Sodium Uranete	Miltrate and Wash Water	Jupure Filter Cake	Ash from Conversion	Ununtin Solutions	Total Tr.	parecoper 741	TEX TELEOVATED	116 Boovered	1E Broovered	Pamp of Material	
						To Laboratory as Scuples	To Contaminated Waste	Dumped by Davelopment Dept.	To Contaminated Storage	To Conteminated Storage	To Contaminated Storage	To Contaminated Storage	Fed and in Storage	unaccounted and Piping	To Fure Storage Coded Chem.	As Samples to Laboratory	Fed to Cascade	Maposition	
							605. gals.		13215. gals.	2684.7 Lbs.	460.55 lbs.	3089. gals.	783.93 lbs.	21.07 lbs.**	143.30 lbs.	10.62 lbs.	640.63 lbs.	Lbs. or Gals.	
-117.80 lbs.	986.71			868.91		6,25***	2,52	n1.1	105.39*	63.02*	137,29	2.88	530.12	14.25**	96.89	7,19	433.23	Lbs. Th	
				100.00		.72	.29		12.13	7.25	15.80	. 33	61.01	1.64	11.15	.83	49.86	or ala	Percent Distribution
					Charles of the same of the sam		2240002	The state of the s	2284667	8880287	2249543	2256751		2268506	22/4082	2264305	2264305	Porcent "7"	Weighted Average Ass

No analysiz available on about one quarter of the containers and the accuracy of about one half of the filtrate analysis which are available ore questionable.

All weighted win average assays except ${
m TF}_6$ fed are either extimated or contain estimated figure.

Chaired lost in transferring Batch 12 from small oglisters to one large cylinder for sampling and feeding.

wan Katimated.

ARLE 2

Amount of Whato Red to Recovery and Development up to December 3, 1946

(Excluding Carbon and Oils)

Spinardel and Class	Gel. or libe.	liber 278	Botinated "T" issor
Swater Medier Class A	5313 gal.	782.17	2268006
3 3 3 B	3915 gel.	91.56	2250004
· 4 9 \$ \$	4214 gal.	38.46	2280002
a a a b	550 gal.	10.45	2460006
n n n n	6391 221.	<u> </u>	2270001
· · · · · · · · · · · · · · · · · · ·			
Rotal to Recovery	20,384 gal.	969.94 lbs.	
Development Department	5,099 gal.	266, 32 1ha	
Sotal "Hater Medic" Intering a Recovery Pressa Enventory 12-31-46	25,483 gal. 1,624	1236.35 lbs. 339.87 lbs.	
Met Reworked to Recover	ry 24,059 gal.	896.49 lbs.	
Process 12-31-45 816 & 616 Mixture	EMPONY SOLO		
Class B	686 lbs.	268.81.1bs.	
Net lbs. *T Charged to Re-working		1165.30 lbs.	
Conversion & T308 Furs Storage		178,59 lbs.	
Inventory 12-31-46			
Not a To in material (I to Representing	narged	986.71 lbs.	

TABLE S

By-products	
Including	
1946	
cember 31	
n hand De	
Materials o	
Waste	
r" Bearing	
200	

	Reworked Material	Franched Material (Estimated 12-31-46)		
Name of Meterial	location	Ibe, or Gal,	Ibs. "I"	Av. "X" An-
Filtor cake from MFL oil	Development Dept.	51.8 lbs.	17,24	2281326
Filter cake from 6-2144 oil	2	190。 1bs.	756°9	2257734
Contaminated C=2144	Contaminated Storage	800 156,	2,000	
Contaminated MFL	62	2000 1b#°	000°9	
Contaminated oil (G-2144 & Mech) Oil Recovery	Oil Recovery	1748.9 1bs.	6,277	
T30g from Carbon	Development Dept.	2163.75 lbs.	1630,	3358505
Raw Filter cake (Recovery)	Contaminated Storage	2684.7 lbs.	20,58	8880287
Ash (from conversion)	Contaminated Storage	460.55 lbs.	137,29	221,951,3
Nater media, incl. filtrate,				
caustic solutions	Contaminated Storage	35,438 gal.	1033,96	5278175
Carbon and Alumina	Contaminated Storage	272,090.1bs.	21,997.47	
Carbon and Alumina	Development Dept.	7,525.8	1,412,79	
TF racovered from Converters Development Dept.	relopment Dept.		1,05	2290001
Remorked Products in Process at "water media" and crecovery conversion, decontamination, T ₃ O ₈ pure Total Estimate of "T" in Waste Materials (12~31~46)	water media" and oil dration, T ₃ O ₈ pure atorage terials (12~31~46)		585.95	

Note: All "X" assays estimated or contain estimated figures.

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